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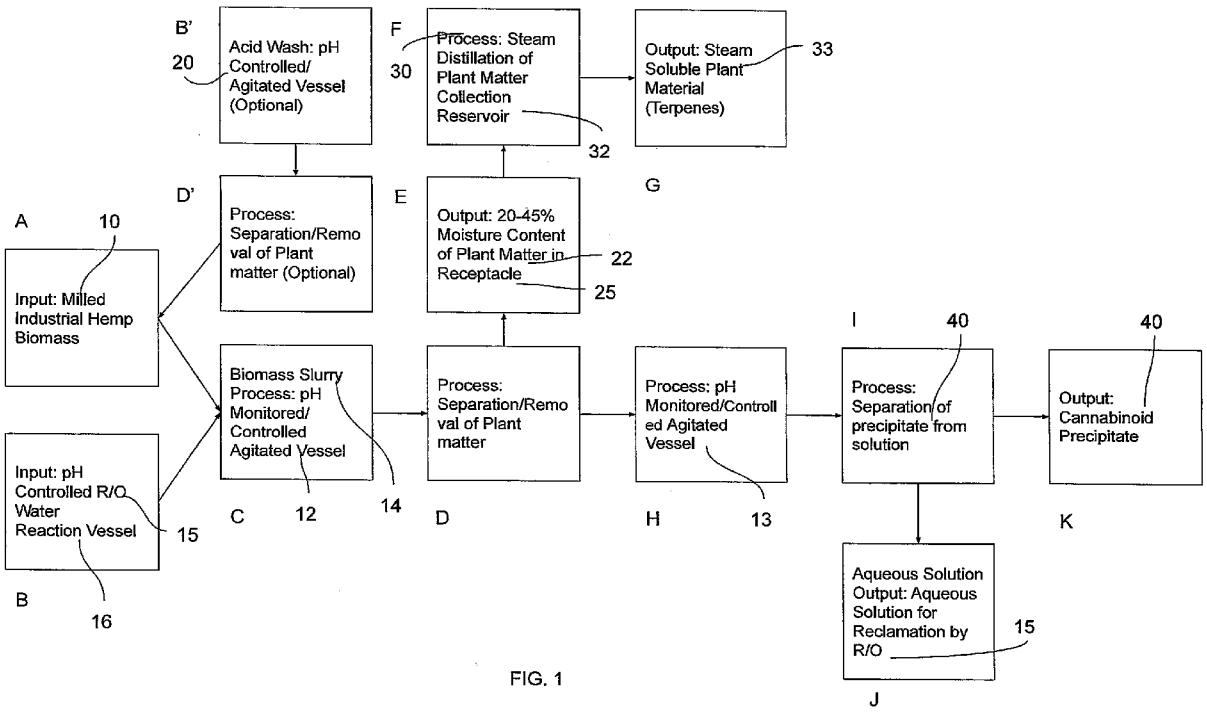


FIG. 1

(57) Abstract: An environmentally friendly and ecologically safe method which is scalable for the aqueous extraction and high volume production of cannabinoids from industrial hemp (cannabis sativa) biomass without the use of hazardous solvents and chromatography techniques. The process improves the efficiency of the extraction process, reduces energy consumption, eliminates fire and explosion hazards and enhances the yield and the purity of the extract for downstream processing.

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[Continued on next page]

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CANNABIS SATIVA AQUEOUS CANNABIDIOLIC ACID EXTRACTION

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to U.S. Provisional Patent Application No. 62/700,710 filed July 19, 2018, the entire disclosure of which is incorporated herein by reference.

FIELD OF THE INVENTION

[0002] The present invention relates generally to the extraction and isolation of natural products from plants. Specifically, the present invention relates to a method for the extraction of cannabinoid compounds from industrial hemp. More specifically, the present invention relates to an ecologically-safe and efficient method for the extraction of cannabidiolic acid (CBDa), or any acidic form of cannabinoids, from the *cannabis sativa* hemp plant.

BACKGROUND OF THE INVENTION

[0003] The extraction, separation and isolation of natural products from their host organisms, be they plants, animals or micro-organisms, is a technically challenging task. Over time, many natural products have been isolated and studied on a laboratory scale for use in food, pharmaceutical, and human and veterinary medical applications. However, extraction and isolation of these natural compounds from their host organisms even on a laboratory scale frequently requires the application of complex and sensitive processing techniques and the use of sophisticated and expensive equipment. Methods which have proven successful for isolating new compounds include liquid-solid chromatographic techniques such as high-performance liquid chromatography, thin layer chromatography, nuclear magnetic resonance processes and Fourier-transformed infrared spectroscopy, just to name a few. While useful for research purposes, the afore mentioned and related exemplary systems and methods have not been scaled or optimized for commercial production. As an example, most industrial scale plant extractions demand the use of large volumes of solvent, such as hydrocarbons and alcohols, or enormous supercritical CO2 systems. This inherently introduces hazards and limitations revolving around the sheer volume of solvents required to extract the

biomass at required volumes. These limitations are enforced by both environmental and regulatory bodies with strict restrictions on their use and residual solvent. Additionally, cleaner systems such as those using supercritical CO2 require a large footprint and operate under immense pressure, thus imposing their own dangers and limitations. Therefore, strong innovation and design incentives exist to remove, or to reduce, the amount of solvents required to extract natural products from their host organisms by environmentally friendly processes. One clear example of this is in the area of cannabis and the extraction of cannabis biomass and phytocannabinoids from hemp plants.

[0004] During the last half of the twentieth century, as an outgrowth of societal changes which began in the 1960's, concerns over climate change and an **enhanced** environmental awareness, interest has increased dramatically in the use of ecologically safe, naturally occurring and so-called "environmentally friendly" compounds and associated manufacturing practices in various applications. Among such applications, alternative forms of medicine which make use of natural products for the holistic treatment of various medical and psychological disorders are being investigated actively.

[0005] The hemp and marijuana genus *(cannabis)* is a source of natural products known as cannabinoids, now recognized as having beneficial medical and therapeutic applications. Cannabinoids are compounds naturally present in the plant that, depending on the specific cannabinoid, have psychoactive and therapeutic effects on certain medical conditions. C*annabis* includes at least three recognized species: *cannabis sativa, cannabis indica and cannabis ruderalis,* which have been used in various forms since ancient times, including use in Asian herbal medications dating back to 2000 BC, as a food source (seeds), and in fiber production for textiles Clearly, cannabis is one of the more ancient and multifaceted cultivars of man to date.

[0006] Of interest to the medical and scientific community, the *cannabis sativa* species contains high concentrations of several medically relevant cannabinoids, for example: cannabidiolic acid (CBDa), its decarboxylated derivative, cannabidiol (CBD), cannabigerolic acid (CBGa) and other phytocannabinoids. Despite its clear utility across multiple cultures, c*annabis* has received considerable negative publicity over time because of certain undesirable psychoactive compounds and trafficking that has

resulted in the classification of the genera as contraband. Until recently, the illegality of its use on both the state and federal level has limited science and medical researchers' ability to fully investigate cannabis' utility. Recently, the non-psychoactive medicinal and therapeutic properties of the lesser publicized cannabinoids have begun to offer innovative therapies to the medical community in the treatment of chronic pain, inflammation, certain cancers, epilepsy, seizures, and infections. To this end, cannabinoids are currently poised to be a potential combatant against the current opioid epidemic and to provide an alternative to treatment option for chronic ailments for which opiates are the current predominant course of action.

[0007] The extraction of cannabinoids from the native plant is a complex and potentially hazardous process which requires the use of high-pressure supercritical carbon dioxide and/or highly flammable and toxic solvents such as benzene, ethanol, methanol or alcohol. The high solvent to biomass ratio, required for efficient extraction of cannabinoids leads to the presence of potentially hazardous amounts of solvents on site and in operation, which scales linearly as the desired volume of active molecules is increased, ultimately reaching levels that are regulatory and economically prohibitive. As the demand for cannabinoids increases so, too, does the demand for, a more efficient, environmentally friendly, extraction process to reduce or eliminate the quantity of solvents needed for biomass extraction.

[0008] The extraction process is further complicated by the variations in composition content within each individual plant, which may vary significantly. Moreover, current extraction procedures may leave unhealthy by-products in the extract such as waxes, fats, and residual solvents. Conventional processing methods incorporate a process known as "winterization' or alcohol washing to remove these by-products. Winterization requires soaking the extract in alcohol and then freezing it, to cryogenic temperatures, for at least twenty-four hours to separate out the unwanted plant waxes and lipids. The winterization process may be repeated several times to achieve the desired purity of oil. However, each round of winterization is accompanied by an associated loss of cannabinoids, thus reducing the extracts overall effectiveness.

[0009] Cannabidiol (CBD), the decarboxylated form of the endogenous cannabinoid CBDa, has been the subject of considerable historical research, and its

efficacy in medicinal applications is well-established. Research on cannabinoids, outside of CBD and THC alone, is only now expanding with enhanced interest and receptivity in the medical and scientific communities. Decarboxylation of all acidic cannabinoids requires a specific amount of activation energy to perform the reaction. This is generally performed at temperatures between 120°C to 140°C for extended amounts of time, up to two and a half hours in some instances. Current extraction methods require this step to move through distillation for further purification. The efficiency of decarboxylation is in the approximate range of 70-85%. Processes that contain a pathway for isolating the acidic forms of phytocannabinoids are beneficial to supply and support ongoing research and demand for each given molecule.

[00010] In view of the above, it will be apparent to those skilled in the art from this disclosure that a need exists for an improved, environmentally friendly method for extracting cannabinoids from industrial hemp biomass that is scalable and adaptable for commercial production, and which eliminates the use of toxic and explosive solvents, reduces energy consumption, and enhances extract yield and purity. The present invention addresses these needs in the art as well as other needs, all of which will become apparent to those skilled in the art from the accompanying disclosure.

SUMMARY OF THE INVENTION

[00011] To address the needs in the art, in one aspect the present invention provides an ecologically-friendly method for the aqueous extraction of cannabidiolic acid from industrial hemp *(cannabis sativa)* biomass which reduces the number and enhances the efficiency of existing extraction protocols.

[00012] In another aspect of the present invention, an ecologically friendly method for the aqueous extraction of acidic cannabinoids from industrial hemp biomass is provided which minimizes the amount of energy required for and the carbon footprint generated by each process cycle.

[00013] In still another aspect of the present invention, an ecologically friendly method for the aqueous extraction of acidic cannabinoids from industrial hemp biomass is provided which eliminates the use of highly flammable and toxic solvents in any of the biomass extraction steps.

[00014] In yet another aspect of the present invention, an ecologically friendly

method for the aqueous extraction of acidic cannabinoids from industrial hemp biomass is provided which eliminates any process barriers to scalability, thereby allowing batch sizes which are constrained only by the size of a process reaction vessel.

[00015] In another aspect of the present invention, an ecologically friendly method for the aqueous extraction of cannabidiolic acid from industrial hemp biomass is provided which operates at efficiencies in a range of approximately 85% to approximately 99% from cannabis biomass.

[00016] These and other features, aspects and advantages of the present invention will become apparent to those skilled in the art from the following detailed description of preferred embodiments taken in connection with the accompanying drawings and by **refe**rence to the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[00017] Referring now to the attached drawings which form a part of this original disclosure.

[00018] Fig. 1 is a flow diagram of the process of the present invention in accordance with an embodiment.

[00019]

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

GENERAL INTERPRETATION OF TERMS

[00020] Selected embodiments of the present invention will now be explained with reference to the drawings. It will be apparent to those skilled in the art from this disclosure that the following descriptions of the embodiments of the method herein disclosed are provided for illustration purposes only and not to limit the invention as defined by the appended claims and their equivalents.

[00021] Referring now to Fig. 1, a flow diagram or chart is provided to illustrate the steps of an ecologically friendly method for the aqueous extraction of acidic cannabinoids from industrial hemp *(cannabis sativa)*. Following the harvesting of a hemp plant, it is shucked and cleaned to remove dirt and other surface contaminants from the plant's stem, leaves and flowers. The stem, leaves and flowers are also detached from the plant's root structure. As shown in Fig. 1. at step a., the stem, leaves and flowers of the plant are cut or masticated into strips each having a length of

approximately six inches using a cutting blade or other suitable cutting apparatus. The six-inch strips are then cut or milled into uniform segments or pieces, each segment being approximately one cubic centimeter in size which gathered together, form a cannabis biomass 10 for further processing. Typically, prior art processes include a drying step prior to the cutting step noted above. In contrast to the prior art, using the aqueous extraction process of the present invention, the biomass is processed wet, lending to a more rapid extraction from field to extract. This is one of the many advantages of the proposed invention.

[00022] This biomass is introduced into a suitably sized reaction vessel such as an agitated reactor or vessel 12 shown in step c. in Fig. 1. By way of example, an agitated reactor having a 1,600-gallon reactor may be used. However, it is understood that the equipment and processes of the present invention may be scaled up or sized for any level of operation without departing from the scope hereof. For example, a processing system having a 4,000 gallon agitated reactor may be employed with satisfactory results. Regardless of size, the process may be monitored continuously for temperature, composition, pH and so forth as is known in the art. It is important to have the slurry homogenized for the full reaction to take place.

[00023] A measured, preselected volume of a purified, pH-controlled aqueous solution is added to the vessel 12, as shown in Fig. 1. at step b. By way of example, the aqueous solution may be reverse osmotically purified water; indicated as R/0 water 15 at step b. However, other solutions of equivalent composition and purity may be used without departing from the scope of the present invention. Prior to adding the purified aqueous solution to the biomass, the aqueous solution's pH is monitored and carefully controlled via the addition of caustic or bases to the solution to attain a specified pH level. By way of example and not of limitation, caustics which may be used in this step include: sodium hydroxide, potassium hydroxide, sodium borate, sodium tetra borate, and calcium hydroxide. Once the aqueous solution has reached the preselected pH, it is pumped into the reaction vessel or agitated reactor 12 containing the hemp biomass to create a biomass slurry 14.

[00024] Optionally, prior to this step, an acid wash of the biomass is proven to increase efficiency and downstream complexations that arise from the formation of

soaps, treatment of waxes and the removal of gums. The acid washing step (shown as B' in the process flow diagram of Fig. 1) functions as a refining aid and thus increases the overall extraction efficiency. These contaminants have the ability to bind up cannabinoids and increase the amount of necessary downstream processing. With this method, extraction efficiencies of 99% can be achieved from a cannabis biomass. This step is not necessary, but it will increase the efficiency of extraction from the overall biomass. The pH will be driven down by the addition of a suitable acid to remove target contaminants. However, as this reaction occurs it consumes the acid, and as the acid concentration decreases, the pH increases, thus necessitating the addition of more acid, a process known in the art as "active mixing". During this period, the pH of the mixture is monitored and controlled at a level in the range of approximately 2.5 to approximately 3.5, and preferably in the range of 2.5 to 3.0. The pH is slowly decreased to 3.0 and readjusted to this pH by the slow addition of HCL or other suitable acids. Once the biomass is washed with the acidic solution (ideally but not limited to hydrochloric acid), the aqueous solution is separated and the biomass is processed further for extraction of cannabinoids as will be described below.

[00025] An optional acid solution 20 may be added to the purified water 15 to control the desired pH of the water. In addition, the water temperature may be adjusted, by heating it preferably in the range of from room temperature to the melting point of cannabinoids, approximately 20° C to approximately 68° C.

[00026] Referring now to Fig. 1. step c., after the aqueous solution is added to the biomass in the reaction vessel, the mixture is agitated for a preselected time period, preferably for at least fifteen minutes, to ensure that the slurry is homogenous. The pH will be driven up by the addition of a suitable base to deprotonate the target cannabinoids. However, as this reaction occurs it consumes the base, and as the base concentration decreases, the pH decreases, thus necessitating the addition of more base, again referred to as "active mixing" as noted above. During this period, the pH of the mixture is monitored and controlled at a level in the range of approximately 8.5 to approximately 10.0, and preferably in the range of 9.0 to 9.5. The pH is slowly increased to 9.5 and readjusted to this pH by the slow addition of NaOH or other suitable bases. This pH is necessary to achieve deprotonation of majority of the acidic

cannabinoids. At this step, the process is not temperature dependent, and the agitation and pH monitoring control is carried out at room temperature. However, this step may also be performed at temperatures above room temperature, but not exceeding the boiling point of water, without departing from the scope of the present invention. Careful control of the agitation time, pH and temperature variables are important to prevent the extraction of unwanted compounds and/or lysing of vegetative cells. Extracting at temperatures above the melting point of decarboxylated cannabinoids, approximately 70° C, increases the overall extraction efficiency.

[00027] The next step in the extraction method of the present invention, which is separation of the solid biomass plant matter and the aqueous solution into two components for further processing of each, is shown at D. in the flow diagram of Fig. 1. The separation step may be performed by passing the biomass-containing solution through a filtering mechanism such as a screen apparatus or a cartridge filter or by processing the solution in a centrifuge, such as a decanter or decanting centrifuge, a disk stack centrifuge, through a plurality of in-line polishing filters following the processing in a decanting centrifuge or other similar apparatus as is known in the art. At this point, the separated biomass indicated by numeral 22 at step e. has a moisture content in a range of approximately twenty percent (20%) to approximately forty five percent (45%), and it is placed in a suitable receptacle 25, by way of example and not of limitation, an auger pipe of appropriate size for additional processing or disposal.

[00028] At step f. further processing, by way of example and not of limitation, steam distillation of extracted biomass will remove any residual steam-liberated molecules from the plant matter. The steam 30 is condensed, and the condensate is collected in a reservoir 32 where the steam-soluble biomass material 33 is separated from the condensate by conventional separation processing, for example by passing it through a disk stack centrifuge and then collected at step g. The steam-soluble biomass material may include terpenes and other commercially usable substances which could be sold separately or recombined with processed CBD to obtain a full profile product. The water is purified and recycled for use in additional processing, and the dry plant matter remaining, which contains cellulose, proteins and sugars, may be pelletized for use in various applications such as animal feed, bedding, absorbents,

compost, fertilizer and building materials. The entire process is designed to be waste-free.

[00029] Referring again to Fig. 1, the pH of the aqueous solution 15 remaining in the reaction vessel after separating the plant matter 22 therefrom is monitored continuously in step h. and adjusted/controlled by the selective addition of an acid to a reaction vessel 13 as is described above to force precipitation of cannabinoid precipitates, or protonation of cannabinoids, from the aqueous solution. Acids suitable for this application including but not limited to: nitric acid, acetic acid, phosphoric acid, hydrochloric acid, sulfuric acid, hypochlorous acid and per chloric acid. The pH of the aqueous solution is passing through a buffer stage in which the pH swings up and down. The pH may be driven down by the addition of a suitable acid to create a precipitate. However, as the precipitate starts to form, it consumes the acid, and as the acid concentration decreases, the pH increases, thus necessitating the addition of more acid, the actual mixing process discussed above. As noted above, the solution pH is initially in a range of approximately 8.5 to approximately 10.0, and preferably in the range of *9.0* to 9.5. The solution starts to get cloudy at a pH level of approximately 5, and larger aggregates of precipitate begin to appear at a pH level of approximately 4.5. The target pH is approximately 2.0 where the solution leaves the buffer stage. At this point, the extraction of the cannabinoid from the solution in the form of a precipitate 40 is substantially completed, as will be described in greater detail in Step I below. The process is very efficient, yielding precipitate in a range of approximately 92% to approximately 99% from biomass. Preferably, the precipitation process is now stopped. However, important measures must be taken to not exceed the theoretical pKa limit of precipitation, as the variability of cannabinoids in the biomass changes the amount of acidic solution required.

[00030] Fig. 1. Step I illustrates the step of separating the cannabinoid precipitate 40 from the aqueous solution 15. Prior art approaches include settlement and evaporation, particulate filtration and the use of cartridge filter bags and bag filters contained in housings. However, the settlement and evaporation approach is not sufficiently scalable to meet commercial production volume requirements, and the precipitate has properties that rapidly blind most filtration material. Cartridge filter bags

are limited to filtration on a micron scale only and also require frequent manual labor. Disk stack and basket centrifuge techniques work reasonably well but are only approximately 85% to approximately 95% efficient. Decanter centrifuges are able to remove the precipitate with an efficiency of approximately 80% to approximately 90%. However, in accordance with an embodiment of the present invention, a concentrated 100% yield is obtained by using a disk stack centrifuge in conjunction with a cartridge filter or microfiltration system. This allows majority of the precipitate to be separated via centrifugation, ultimately limiting the load requirements seen by the screen-based filtration methodology described below that allows for 100% recovery efficiency.

[00031] The aqueous solution 15 is directed back to a purification source for reuse at Fig. 1 at step j., and the precipitated cannabinoids 40 are collected for further processing, as needed as shown at step k. The cannabinoid precipitate contains, among other compounds, CBN, CBD, CBDa, THCa, THC, THCV, CBC, CBN, and CBG in varying concentrations which are driven by the composition of the plants being processed. As noted earlier, each plant contains varying ratios.

[00032] The cannabinoid precipitate is constituted mostly of cellular debris and oil which requires further processing to remove contaminants and further purify the extract. From this point supplementary pathways may be taken to isolate each given molecule. By way of example but not limitation, the carboxylic acid groups may be eliminated via heating at a preselected temperature for a specific time period in an agitated, jacketed reactor as is known in the art. Optimum temperature and time parameters have been identified to be approximately 100° C for approximately two *(2)* hours, which leaves a cannabinoid-containing oil with the carboxylic acid removed. The various cannabinoids may be separated from one another via fractional distillation techniques and associated apparatus, which have demonstrated output volumes on the order of five (5) gallons per minute, a significant improvement over prior art processes which, at best, deliver approximately one liter per hour.

EXAMPLES

Trial 1

[00033] A 0.000001M NaOH standard solution with tap water was prepared and

measured the pH to find it was 5.25. More 0.1M NaOH was added to make the standard solution pH 8.5, the molarity is .001M. 100mg ground hemp flower *w*as weighed and added it to 20mL of the standard solution and stirred with a stir plate and stir bar, continually checking the pH and adding standard solution as needed to maintain the pH at or above 8. The pH could not be increased above 8 so the standard solution was adjusted to pH 10, a new molarity of 0.0156M. Even so, the pH never reached 8 - even after twenty minutes the highest pH was 7.5. Two .25mL aliquots of 0.1M NaOH were then added to get the pH to 8, then 9 where it maintained for 5 minutes with no additions. The total extraction time was 30 minutes where 10 minutes was spent at the ideal pH. The biomass was filtered out and the filtrate was stirred with a stir bar and stir plate. To the biomass filtrate 1.0 uL titrations of 2.OM HCl were added and changes in pH and appearance were noted. The clear yellow filtrate turned cloudy at pH 5.5 but the pH was brought all the way to 2.5. There was no change in appearance from pH 5.5 to PH 2.5. The precipitate was filtered but all the precipitate went straight through the filter paper. The precipitate solution was then poured onto a watch glass and dried covered for 3 days in a hood. The result was 0.03g +/- 0.01g which is a 30% yield. The precipitate was reconstituted with 3mL HPLC grade methanol for further testing. Dried precipitate is stored at room temperature protected from light. There were three trials that used the technique described below, which produced the best purity, not the best percent yield. This trial has a slower addition of base during the extraction and acid during precipitation which pro*v*ed better for purity sake.

Trial 2

[00034] A pH 7 rinsing solution was prepared using tap water and 0.1M NaOH. A 0.1M solution of HNO3 was prepared staring with 70% HNO3. 2 mL pH 7 rinsing solution was added to the reaction beaker and 140mg ground hemp flower *w*as added and then stirred with a spatula instead of a stir bar. 20-40ul aliquots of 0.1M NaOH were added to reach and maintain pH 8.5. Total extraction time was 15 minutes and the amount of time spent above pH 8 was 10 minutes. The biomass was then filtered and 40 UL aliquots of the 0.1M HNO3 solution were added to the filtrate. Changes in pH and appearance were noted. The clear yellow solution turned cloudy at pH 4.5 but the pH was brought all the way to 3, though there was no change in appearance from pH

4.5 to 3. The volume of the precipitate solution was noted an equal volume of cold hexane was added and shaken in a separation funnel vigorously for 2 minutes. The two layers were separated and the hexane layer was dried on a watch glass. The result was 0.01g precipitate (7.14% yield) that was 85.32% pure CBDa, derived from HPLC analysis. Thus, the percentage of the plant material that was extracted as CBDa was 8.125%. The bottom aqueous layer was dried in an oven for 6 hours in an oven at 65C and then for 3 days in a hood. The resulting mass was 0.03g that was 1.26% pure CBDa per HPLC analysis, so a portion of the precipitate was not taken up by the cold hexane and was lost in this bottom layer.

Trial 3

[00035] A solution of 3,000 mL of distilled water (4.95 pH starting) and 60g of Andres Hemp was prepared and was washed with acid (HCL) under the following conditions:

50° C

pH 3.0 held 3,000mL

Post Acid Concentration (Flower)

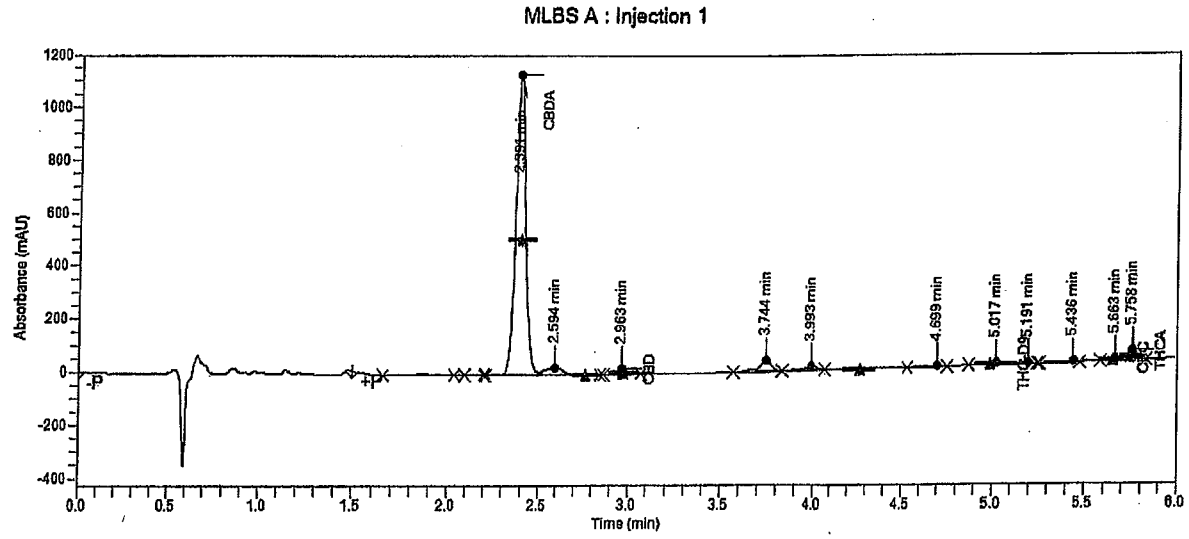
Acid Wash Concentration: (wash water)

The mixture was then washed with a 3,000 ml caustic solution at a pH of 10. A slightly elevated temperature between 80°C and 87°C was observed toward the end of the wash cycle. A precipitation step was initiated with the pH of the HCL maintained at 3.0 and the temperature held in the range of approximately 39°C-40°C. It was observed that even as the solution cooled, the supernatant remained hazy and precipitation continued as the solution cooled down. The supernatant sample smelled strongly of sulfur. It was partially dried in an oven overnight. A portion of the sample was dark and flaky in texture and a portion was still moist after the overnight drying process.

ANALYTICAL TABLES

ANALYTIC TABLE 1

Sample Name	MLBS A **Pre-extract hemp biomass**
Batch Group/Name	Farm Testing
Acquisition Method	PE Flower Method_150mm_original_adjustable
Processing Method	PE Flower Method_150mm_original_adjustable



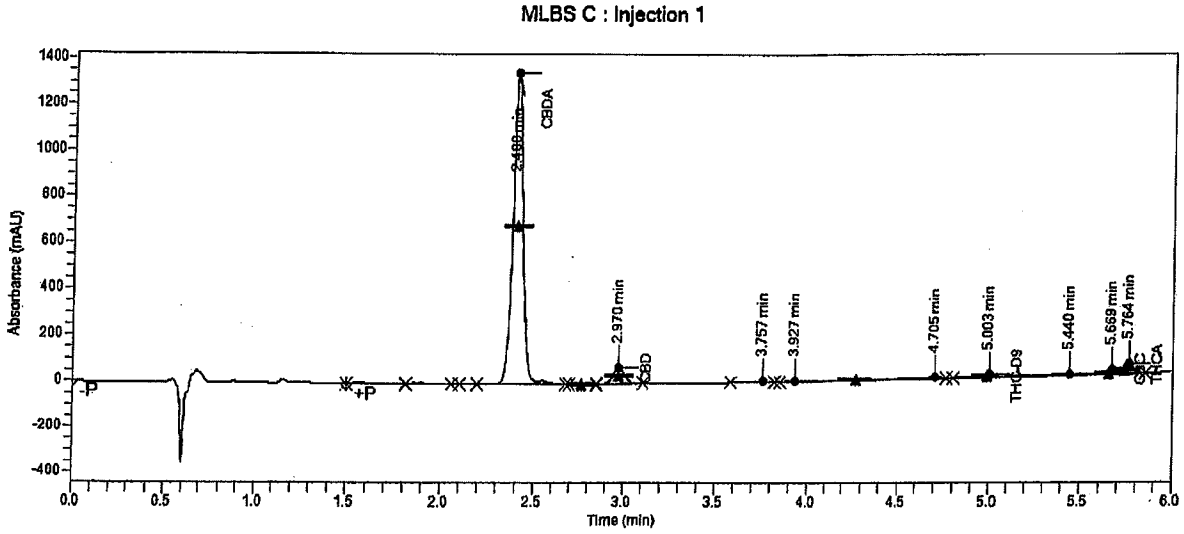
ANALYTIC TABLE 1 (continued)

Peak #	RT (min)	Component Name	Area	Height	BL	Final Amount	Units
	1.577	CBDV					
1	1.701		5,385.2	1,180.4	BV		
2	1.867		18,696.7	4,495.0	VB		
3	2.149		1,336.9	401.6	BB		
	2.245	CBGA					
4	2.391	CBDA	4,330,124.6	1,134,913.1	BE	101.6931	µg/mL
5	2.594		101,929.0	28,481.7	EV		
6	2.762	CBG	3,765.6	975.2	VB	0.2634	µg/mL
	2.806	THCV					
7	2.963	CBD	82,779.5	19,070.3	BB	3.4272	µg/mL
8	3.744		177,688.6	40,323.9	BB		
	3.874	CBN					
9	3.993		63,259.7	13,581.5	BB		
10	4.699		14,090.2	3,810.5	BB		
	4.712	THC-D8					
11	5.017	THC-D9	29,430.9	6,469.9	BV	1.3965	µg/mL
12	5.191		19,168.0	5,924.9	VB		
	5.329	THCA-A					
13	5.436		7,025.1	1,761.2	BB		
14	5.663	CBC	22,764.9	7,244.4	BV	0.4194	µg/mL
15	5.758	THCA	124,181.7	37,819.3	VB	3.0030	µg/mL
Total			5,001,626.6			110.2026	

10.1% cannabinoid concentration

ANALYTIC TABLE 2

Sample Name	MLBS C **Post Acid Wash Hemp Biomass**
Batch Group/Name	Farm Testing/
Acquisition Method	PE Flower Method_150mm_original_adjustable
Processing Method	PE Flower Method_150mm_original_adjustable



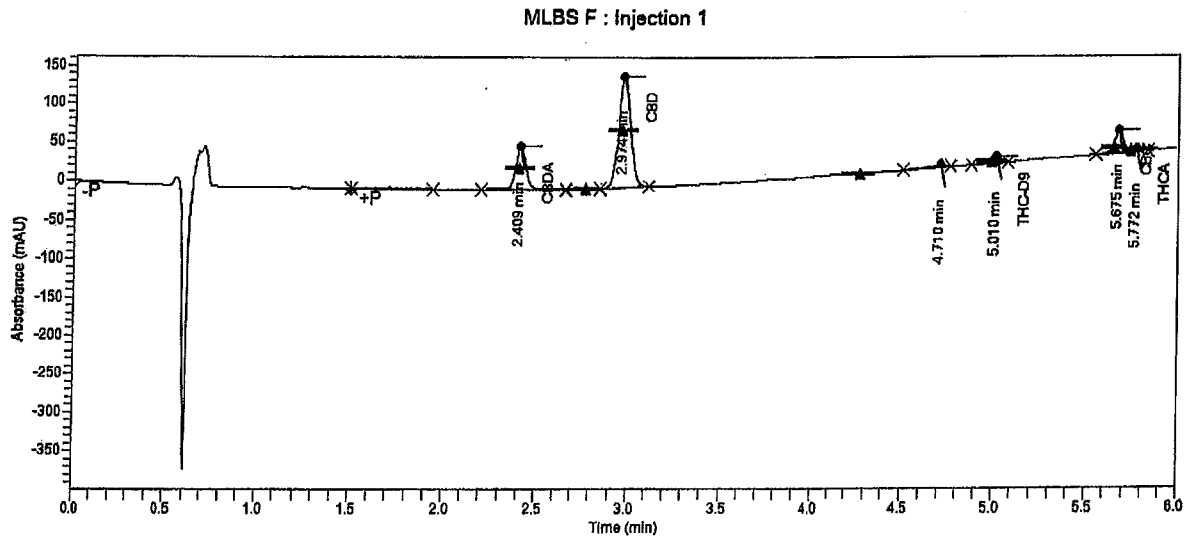
ANALYTIC TABLE 2 (continued)

Peak #	RT (min)	Component Name	Area	Height	BL	Final Amount	Units
1	1.560		24,455.5	7,075.5	BV		
	1.577	CBDV					
2	1.702		8,422.8	2,070.7	VB		
3	1.872		24,504.7	5,937.1	BB		
4	2.147		959.7	334.9	BB		
	2.245	CBGA					
5	2.400	CBDA	5,098,525.6	1,348,751.3	BB	119.7368	µg/mL
6	2.770	CBG	6,183.1	1,630.7	BB	0.3617	µg/mL
	2.806	THCV					
7	2.970	CBD	300,268.7	68,283.3	BB	12.3208	µg/mL
8	3.757		10,344.2	2,462.7	BB		
	3.874	CBN					
9	3.927		3,708.2	940.0	BV		
10	4.705		24,232.7	6,688.9	VB		
	4.712	THC-D8					
11	5.003	THC-D9	26,585.0	7,033.7	BV	1.2735	µg/mL
	5.329	THCA-A					
12	5.440		13,874.0	2,702.4	VV		
13	5.669	CBC	54,779.3	16,702.2	VV	0.9879	µg/mL
14	5.764	THCA	130,539.3	39,973.8	VB	3.1555	µg/mL
Total			5,727,382.8			137.8361	

11.9% cannabinoid concentration

ANALYTIC TABLE 3

Sample Name	MLBS F **Post Caustic Extraction Hemp Biomass**
Batch Group/Name	Farm Testing/.
Acquisition Method	PE Flower Method_150mm_original_adjustable
Processing Method	PE Flower Method_150mm_original_adjustable



ANALYTIC TABLE 3 (continued)

Peak #	RT (min)	Component Name	Area	Height	BL	Final Amount	Units
1	1.556		1,337.8	220.9	BV		
	1.577	CBDV					
2	1.707		9,680.0	1,382.1	VB		
	2.245	CBGA					
3	2.409	CBDA	208,435.0	55,388.4	BB	4.9066	µg/mL
4	2.774	CBG	7,919.3	1,906.8	BB	0.4323	µg/mL
	2.806	THCV					
5	2.974	CBD	638,730.0	145,048.9	BB	26.1611	µg/mL
	3.874	CBN					
6	4.710		13,727.3	3,850.3	BB		
	4.712	THC-D8					
7	5.010	THC-D9	32,981.2	9,212.0	BB	1.5500	µg/mL
	5.329	THCA-A					
8	5.675	CBC	100,252.9	31,036.9	BE	1.7952	µg/mL
9	5.772	THCA	10,742.5	3,777.3	EB	0.2816	µg/mL
Total			1,023,806.1			35.1268	

0.9% cannabinoid concentration

[00036] In view of the foregoing analytical results of the various end product text samples, the aqueous extraction process of the present invention demonstrates its ability to successfully and efficiently yield cannabinoid extracts of high purity. While only selected embodiments have been chosen to illustrate the present invention, it will be apparent to those skilled in the art from this disclosure that various changes and modifications can be made herein without departing from the scope of the invention as defined in the appended claims. Furthermore, the foregoing descriptions of the embodiments according to the present invention are provided for illustration only, and not for limiting the invention as defined by the appended claims and their equivalents.

CLAIMS

What is claimed is:

1. A method for the aqueous extraction of cannabinoids from industrial hemp *(cannabis sativa)* biomass comprising the steps of:

a. cleaning a harvested hemp plant having a stem, a plurality of leaves and flowers and a root structure by removing the root structure from the plant;

b. cutting the stem and the plurality of leaves and flowers of the hemp plant into a plurality of biomass strips, each strip having a preselected length;

c. milling each of the plurality of strips into substantially uniformly sized biomass segments having a preselected size;

d. introducing the uniformly sized biomass segments into a first reaction vessel;

e. adding a purified aqueous solution having a pH level to a second reaction vessel;

f. controlling the pH level of the purified aqueous solution by adding a controlled quantity of a base/caustic material thereto, whereby the pH level is adjusted to a preselected pH level;

g. introducing the purified aqueous solution having a preselected pH level into the first reaction vessel, whereby an aqueous biomass mixture is created;

h. agitating the mixture for a preselected period of time while simultaneously continually monitoring and maintaining the preselected pH level;

i. separating the biomass and the aqueous solution into two components for further processing of each;

j. continuously monitoring the pH of the aqueous solution following separation of the biomass therefrom in step i;

k. adding a preselected quantity of an acid to the aqueous solution in response to a pH value measured in step n. to force a precipitation of cannabinoid precipitates from the aqueous solution;

l. stopping the cannabinoid precipitation at a preselected aqueous solution pH value;

m. separating the cannabinoid precipitate from the aqueous solution; and

n. directing the aqueous solution to a purification source for reuse in step e.

2. The method of claim 1 wherein cutting the stem and the plurality of

leaves and flowers in step b comprises cutting the stem and the plurality of leaves and flowers into six inch strips.

3. The method of claim 1 wherein the milling each of the plurality of strips in step e comprises milling each of the plurality of strips into one cubic centimeter-sized segments.

4. The method of claim 1 wherein the preselected period of time for which the mixture is agitated in step g. is a minimum of fifteen minutes.

5. The method of claim 1 wherein the agitation of step h. is performed at room temperature.

6. The method of claim 1 wherein the preselected pH level in the agitation of step h is in a range of 8.5 to 10.0.

7. The method of claim 6 wherein the preselected pH level in the agitation of step h is preferably 9.5.

8. The method of claim 1 wherein the separation of the biomass and the aqueous solution in step i. comprises passing the biomass-containing solution through a filtering mechanism.

9. The method of claim 1 wherein the separation the biomass and the aqueous solution in step I comprises processing the biomass-containing solution in a centrifuge.

10. The method of claim 9 wherein the step of processing the biomass-containing solution in a centrifuge comprises processing the biomass-containing solution first in a decanter centrifuge and thereafter in a disk stack centrifuge.

11. The method of claim 9 wherein the step of processing the biomass-containing solution in a centrifuge comprises processing the biomass-containing solution first in a decanter centrifuge and thereafter through a plurality of in-line polishing filters.

12. The method of claim 1 wherein the acid added in step o is selected from the group consisting of: nitric acid, acetic acid, phosphoric acid, hydrochloric acid, sulfuric acid, hypochlorous acid and per chloric acid.

13. The method of claim 1 wherein the base/caustic material added in step f is selected from the group consisting of: sodium hydroxide, potassium hydroxide,

sodium borate, sodium tetra borate, calcium hydroxide and any base anhydride.

14. The method of claim 1 wherein the aqueous solution pH value at which the cannabinoid precipitation is stopped in step p is in a range of 2.0 to 4.0.

15. The method of claim 1 further including the step of introducing the biomass into an auger pipe after separating the biomass from the aqueous solution in step i.

16. The method of claim 1 wherein the step of separating the cannabinoid precipitate from the aqueous solution in step q comprises processing the biomass-containing solution in a centrifuge.

17. The method of claim 16 wherein the centrifuge is a basket centrifuge.

18. The method of claim 16 wherein the centrifuge is a decanter centrifuge.

19. The method of claim 16 wherein the centrifuge is a disk stack centrifuge.

20. The method of claim 1 wherein decarboxylating the cannabinoid precipitate of step s comprises heating the cannabinoid precipitate at a preselected temperature for a preselected period of time.

21. The method of claim 19 wherein the preselected temperature is in a range of 90° C to 110° C.

22. The method of claim 20 wherein the preselected period of time is in the range of 1.5 hours to 2.5 hours.

23. The method of claim 1 wherein the cannabinoid precipitate includes CBN, CBD, CBDa, THC, THCA, THCV, CBC, CBGa and CBG; the method further including the step of separating and isolating the CBN, CBD, CBDa, THC, THCA, THCV, CBC, CBGa and CBG from one another.

24. The method of claim 1 wherein the steam-soluble biomass material separated from the condensate in step m includes terpenes, the method further including the step of isolating the terpenes from the condensate.

25. The method of claim 1 further including the step of isolating the cannabinoid precipitate following the decarboxylation of the cannabinoid precipitate in step p.

26. The method of claim 25 wherein the step of isolating the cannabinoid precipitate comprises isolating the cannabinoid precipitate via fractional distillation.

27. The method of claim 1 further including the step of drying the cleaned hemp plant after the step of cleaning in step a.

28. The method of claim 1 further including the step of applying a pretreatment acid wash on the hemp plant biomass prior to step g.

29. The method of claim 1 further including the step of heating the aqueous solution used in the acid wash.

30. The method of claim 29 wherein the step of heating the aqueous solution used in the acid wash comprises heating the aqueous solution to a temperature in a range of 20°C to 68°C.

31. The method of claim 1 wherein the agitation of step h is performed in a temperature range of 75°C to 85°C.

32. The method of claim 1 further including the step of decarboxylating the cannaboid precipitate.

33. The method of claim 1 further including the step of initiating a steam distillation whereby any steam-soluble biomass material is extracted.

34. The method of claim 33 further including condensing the steam to form a condensate.

35. The method of claim 34 further including the step of collecting the condensate in a reservoir.

36. The method of claim 35 further including the step of separating the steam-soluble biomass material from the condensate.

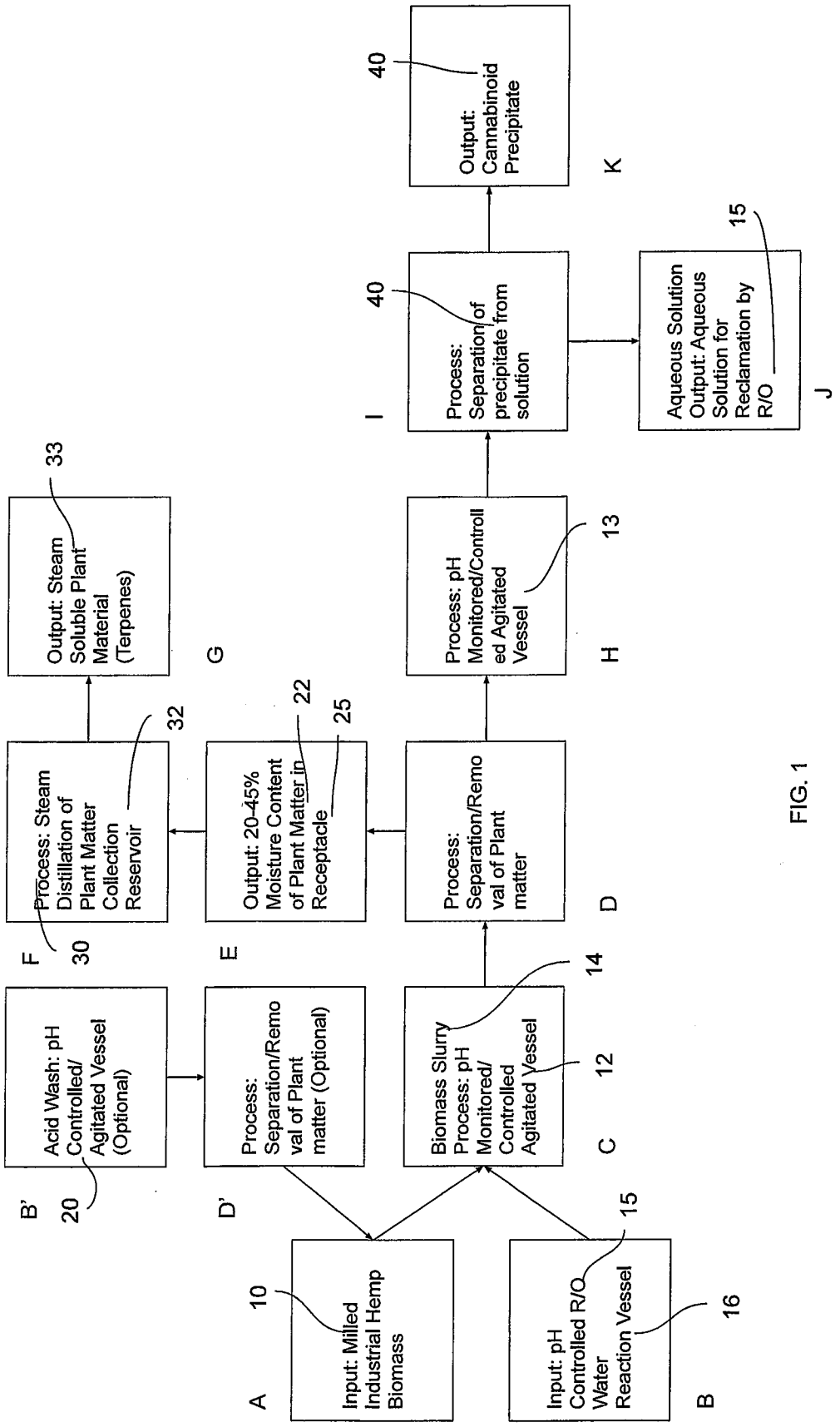

A
Input: Milled Industrial Hemp Biomass
10
B
Input: pH Controlled R/O Water Reaction Vessel
15
16
B'
Acid Wash: pH Controlled/ Agitated Vessel (Optional)
20
C
Biomass Slurry Process: pH Monitored/ Controlled Agitated Vessel
12
14
D'
Process: Separation/Removal of Plant matter (Optional)
D
Process: Separation/Removal of Plant matter
E
Output: 20-45% Moisture Content of Plant Matter in Receptacle
22
25
F
Process: Steam Distillation of Plant Matter Collection Reservoir
30
32
G
Output: Steam Soluble Plant Material (Terpenes)
33
H
Process: pH Monitored/Controlled Agitated Vessel
13
I
Process: Separation of precipitate from solution
40
J
Aqueous Solution Output: Aqueous Solution for Reclamation by R/O
15
K
Output: Cannabinoid Precipitate
40

FIG. 1